A TECHNIQUE FOR THE METALLOGRAPHIC PREPARATION OF LEAD

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ABSTRACT

A technique is described which enables lead to be metallographically prepared to reveal the true microstructure with good edge retention. The technique consists of a series of mechanical grinding and polishing steps each of which is followed by a chemical polish. The depth of the deformed metal layer, due to grinding, was measured as well as the rate of metal removed by the chemical polish. The technique can be used to achieve relatively good results in a short period of time.

INTRODUCTION

The purpose of this paper is to present a relatively rapid and easy technique for the metallographic preparation of pure lead. Due to the extreme softness of pure lead, it is difficult to obtain the true microstructure by mechanical polishing techniques. However, it is also difficult to obtain the microstructure by chemical or electrochemical techniques without severely rounding the edges of the sample. The technique to be described uses a combination chemical-mechanical polishing procedure. It may be used for a very rapid preparation to look only at the bulk microstructure or to obtain a relatively flat edge, not both. The technique can also be used, if very carefully applied, to obtain the true microstructure and still retain relatively flat edges.

The technique was developed using samples which had been welded to enable the true microstructure to be more rapidly recognized. Due to the low recrystallization temperature of lead, about 0°C, it is not always easy to tell if the structure observed represents the bulk material.

PROCEDURE

The sample should be sectioned as gently as possible and mounted in a reasonably soft, room-temperature-curing resin. We used Fiberlay, P-18 coating resin (Fiberlay, Inc.) the hardness of which can be controlled by the amount of hardener added. Other similar resins such as Klearmount (Vernon-Benshoff Co., Inc.) might also be used.

The following steps were used for grinding and polishing the lead samples:

1. Grind on 600 grit SiC with wheel moving to remove deformation due to sectioning or rougher grinding steps. Use a light pressure. Chemically polish using a cotton swab and light pressure, until the metal appears shiny. The polishing solution of 50 ml of hydrogen peroxide.
2. Hand lap on 600 grit SiC (still wheel) to get mount and sample at the same level. Chemically polish as in 1.
3. Hand lap on 600 SiC until scratches appear on the metal, clean the sample and continue hand lapping using 6 μ diamond paste on silk over glass. Chemically polish.
4. Rough polish with 6 μ diamond on Metcloth with a slowly moving wheel. When the appearance of lead on the wheel is uniform, stop the wheel and hand lap briefly. Chemically polish.
5. Fine polish with 0.3 μ Al_2O_3 on billiard cloth and chemically polish by immersion.
6. Final polish with 0.05 μ Al_2O_3 in dilute sodium acetate on Microcloth and chemically polish by immersion. The sample is etched using a solution of 9 g of ammonium molybdate, 15 g of citric acid, and 80 ml of distilled water (30 sec to 1 min). This solution should be allowed to age for 1 hr before use.

The chemical polish is never used longer than 2-5 sec at a time – the sample must be immediately rinsed under distilled water and quickly blown dry.

RESULTS

The appearance of the sample through several of the preparation steps was followed and the same area photographed for comparison.

These show the appearance of the deformed metal as it is slowly removed to reveal the bulk structure. Figure 1 shows the appearance of the weld sample after a 600 grit SiC grind using a moving wheel. Figure 1a shows the appearance as ground. Figure 1b is the same area after a 5-sec chemical polish (etched using a 5 % HNO_3, 10% HOAc solution). Figure 1c shows the sample after an additional 5-sec chemical polish. The decrease in the number of imbedded SiC particles is evident. The crack is a joint and weld metal is above it.

The sample was further chemically polished to remove the SiC particles and then ground through 6 μ diamond on silk.

Figure 2 shows the appearance of the sample after polishing with 6 μ diamond on Metcloth. Figure 2b is the sample after chemical polishing and etching. There is still no obvious difference between weld and parent metal.

Further polishing removes more and more of the deformed metal; evidenced by the decrease in small grains present. Figures 3a and 3b show this effect after polishing with 0.3 μ Al_2O_3 and 0.05 μ Al_2O_3, respectively. In Figure 3b the microsegregation of impurities, or microcoring, of the weld is beginning to become apparent.

After further polishing and etching the final microstructure is revealed. A low magnification photograph of the sample, Figure 4a, shows the flow lines in the base metal and the columnar, cored grains of the weld. Figure 4b shows the appearance of the parent metal.

The appearance of a different lead weld, similarly prepared, is shown in Figure 5a. The weld structure is shown more clearly in Figure 5b.

The ability to preserve the edge while using this technique is shown in Figure 6. However, for good edge preservation it may be necessary to go to finer diamond on silk and it is always necessary to stay on the silk on glass until evidence of deformed metal is a minimum.

Measurements of the depth of the deformed metal were made microscopically after grinding and then chemically polishing to remove metal until the true microstructure was obtained. A sample ground on 600 grit SiC with a moving wheel had to have 45-48 μ of metal removed before most of the flowed metal was gone.

If the sample were hand lapped on 600 grit SiC, the deformation was only 32-34 μ deep.

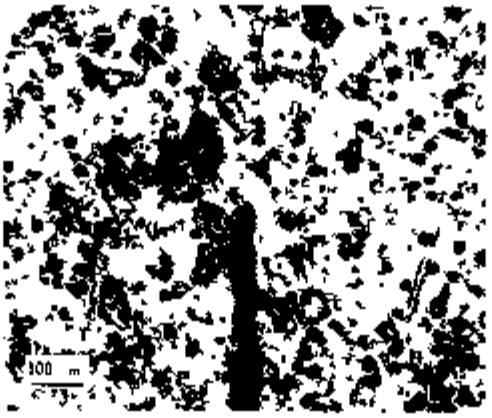
DISCUSSION

In general each mechanical step in the preparation is followed by a chemical polish. The chemical polishing procedure should be carried out in 2-5 sec increments and proceed only to the point where the height difference between the sample and the mount can be readily eliminated by the next step. Approximately 2-4 μ of the metal are removed by immersion in the polishing solution and about twice that is removed by swabbing for 2-5 sec.

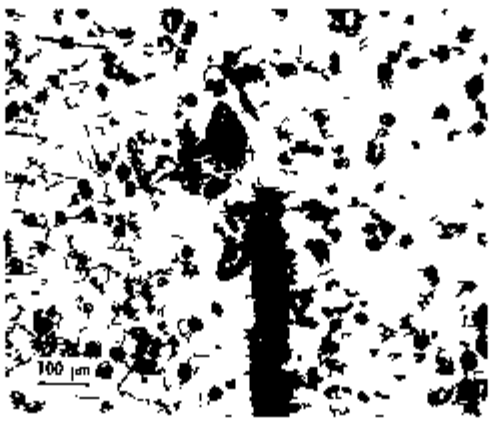
The time required may vary from about one hour to one day depending on what is desired. A scratch free, true microstructure with good edge retention will take very careful work.

The biggest problem with the technique is staining and uneven attack during rinsing. It is imperative that the peroxide be rinsed away rapidly and then the sample dried immediately on removal from the water.

Figure 1a Pb as ground, 100X. (Reduced 63%)

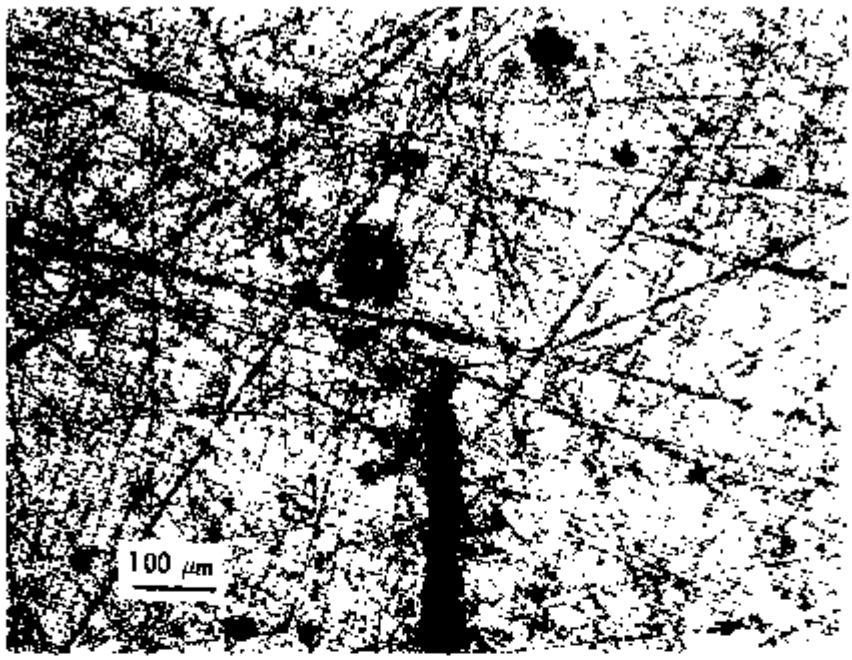


(b) The same area as (a) after a 5-sec chemical polish [$(NH_4)_2 MoO_4$, citric acid etch], 100X (Reduced 63%)

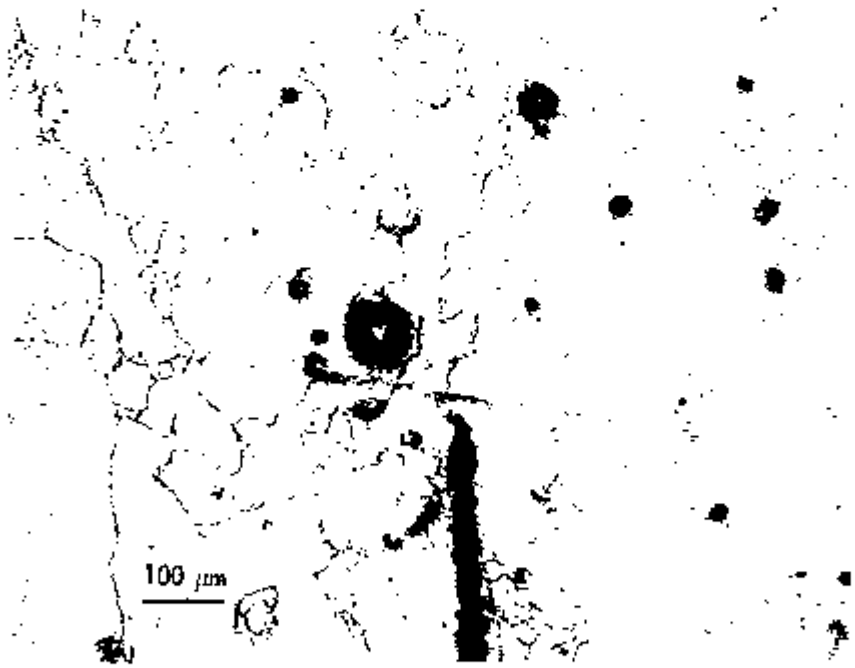


(c) The same area as (b) after an additional 5-sec chemical polish [same etch as (b)], 100X (Reduced 63%)

Figure 1. Photomicrographs of lead weld sample after grinding on 600 grit SiC

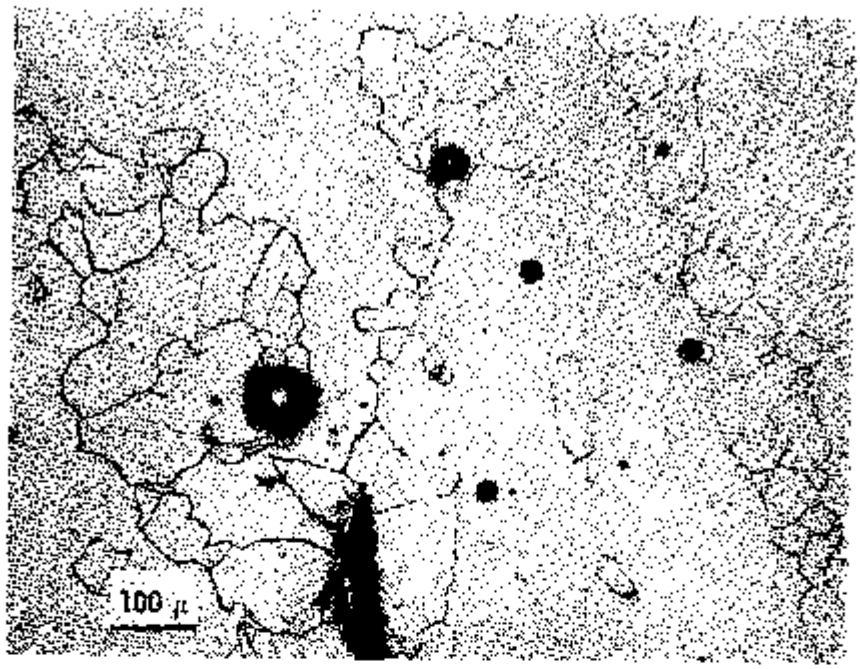


(a) Same area as Figure 1; as polished; 100X.

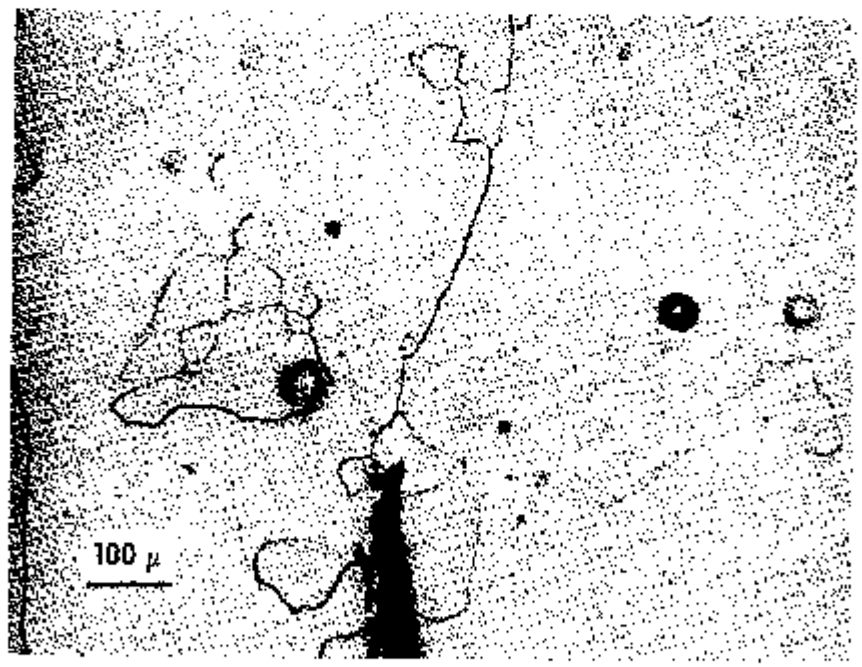


(b) Same area after a 5-sec chemical polish (HOAc, HNO_3 etch); 100X.

Figure 2. A lead weld sample after rough polishing with a 6 μ diamond on Metcloth.

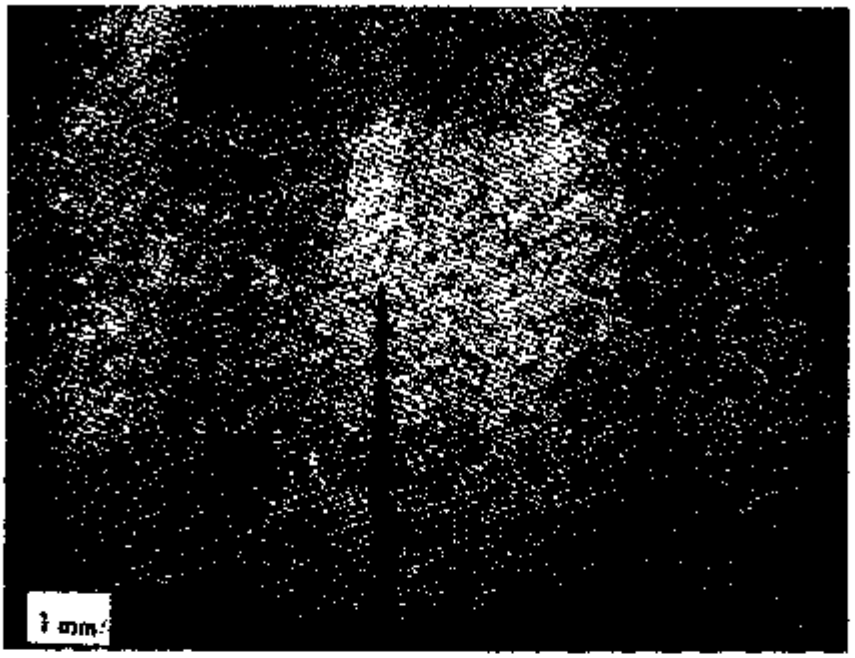


(a) Polished with 0.3 μ Al_2O_3 on billiard cloth; chemical polished (HOAc and HNO_3 etch); 100X



(b) Polished on 0.05 μ Al_2O_3 and chemically polished [etched in $(NH_4)_2 Mo_4$, citric acid]; 100X.

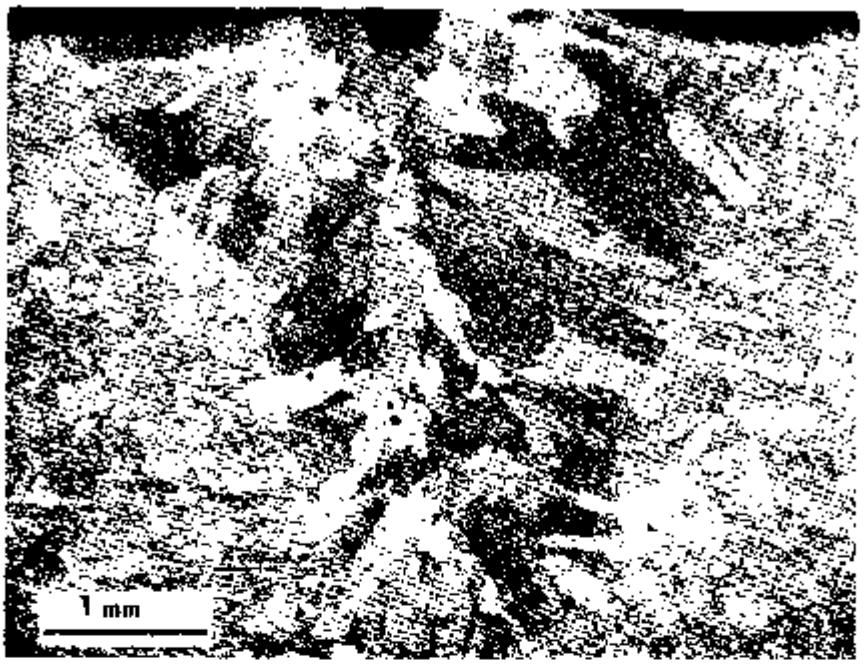
Figure 3. The same area of the lead weld sample as shown in Figures 1 and 2 after further polishing.



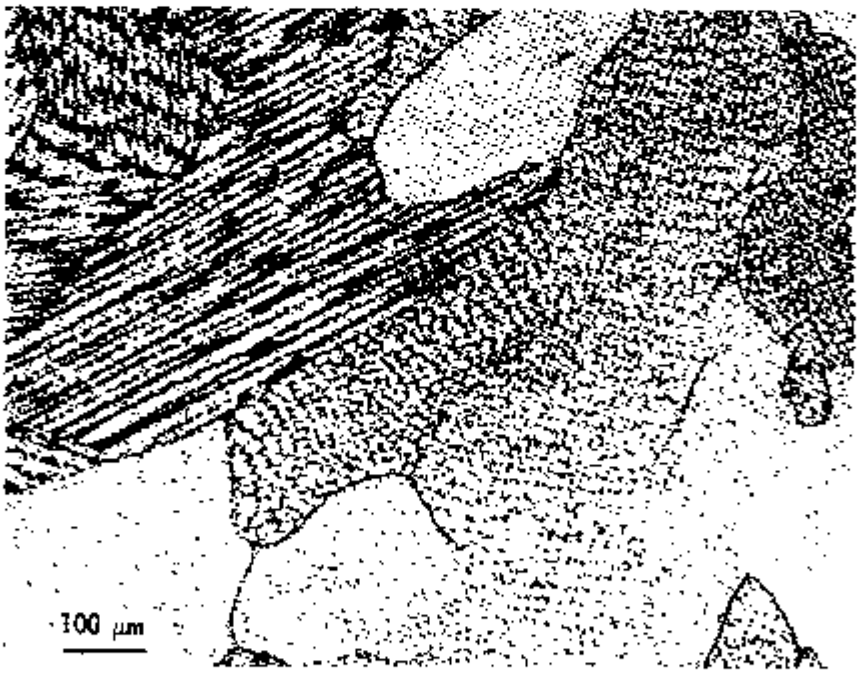
(a) Whole sample at low magnification [$(NH_4)_2 MoO_4$ etch]; 16.5X.

(b) Parent metal; 100X.

Figure 4. Final microstructure of the lead weld sample.



(a) Weld sample at low magnification [$(NH_4)_2$ MoO_4 etch]; 20X.



(b) Weld metal; 100X.

Figure 5. Appearance of the weld in a different sample.